

June 1, 1976

ENDOSULFAN RESIDUE ON THE OUTER LEAVES OF HEAD LETTUCE

IMPERIAL COUNTY, CALIFORNIA - JANUARY 1976

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INTRODUCTION

Endosulfan (Thiodan) is a chlorinated hydrocarbon insecticide-acaricide currently registered for use on a variety of fruit, vegetable and field crops. The major use of the chemical in California is on head lettuce and tomatoes.

In 1975, 173,854 pounds of endosulfan were used to treat 182,277 acres of California head lettuce. The chemical is formulated in 3 to 5% dusts, emulsifiable concentrates containing 2 to 4 pounds endosulfan per gallon and in combination formulations with other pesticides. Acute oral and dermal LD₅₀ values developed with female rats are 18 and 74 mg./kg. respectively. Mammalian toxicity results from nervous system stimulation. There is no specific antidote.

Technical endosulfan is a mixture of two steric isomers: the alpha isomer, or endosulfan I, which constitutes approximately 60% of the technical material, and the beta isomer or endosulfan II. A third more persistent residue, endosulfan sulfate, accumulates in foliage as a result of plant metabolism.

The preharvest interval for endosulfan application to head lettuce is 14 days and the residue tolerance is 2 ppm total for endosulfan I, II and endosulfan sulfate.

APPLICATION AND SAMPLING

In order to measure the disappearance rates of dislodgeable and penetrated residues resulting from endosulfan application to head lettuce, two treated fields were sampled after application at 24 hour and successively longer intervals. The fields were located near El Centro in Imperial County, California, and were designated fields 1 and 2:

<u>Field No.</u>	<u>Date</u>	<u>Materials in Tank Mix</u>	<u>Endosulfan Applied Per Acre</u>	<u>Spray Volume</u>
1	Jan. 13, 1976	Red Top Thiodan 2 - 1.6 qts; Phosdrin, Thuricide	0.8 lbs.	10 gal/acre
2	Jan. 16, 1976	Rockwood Thiodan 4- 1.6 pts., Nudrin	0.8 lbs.	10 gal/acre

Three samples, each consisting of 80 leaf discs, 2.5 centimeters in diameter, were collected from the wrapper leaves of 80 different heads at each sampling interval.

ANALYTICAL METHODS

The procedure used in the extraction of dislodgeable, penetrated and total endosulfan residues is described in an attachment. Two samples of each triplicate collected per sampling interval were analyzed for dislodgeable and penetrated residues; the third for total residue. The analysis was carried out with a Varian Aerograph Model 680 Pestilizer gas chromatograph operated under the following conditions:

Column - 6' x 2 mm I.D. (glass) of 3% OV-101 on 100/120 mesh Gas Chrom Q with 4.5% OV-210 on 100/120 mesh Gas Chrom Q (mixed 1:1 by weight after coating) operated at 215°C.

Detector - H³ @ 215°C.

Carrier gas - N₂ @ 80 psi (approx. 40 ml/min).

Quantitation was by comparison of standard and sample peak heights matched to within \pm 10%.

RESULTS

The dislodgeable, penetrated and total endosulfan residues observed are given in Table 1. Daily weather observations made in El Centro are given in Table 2.

The decline of residue concentrations on and in head lettuce wrapper leaves results from a combination of dissipation, chemical degradation, and dilution by rapid plant growth. The effects of dilution were probably most evident in Field 2 because it contained young lettuce plants with rapidly developing wrapper leaves. Initial deposition rates of endosulfan I and II were consistent with their proportion in the technical material. Endosulfan I exhibited a more rapid decay than endosulfan II in both dislodgeable and penetrated residues. Dislodgeable residues of endosulfan isomers I and II declined to 10% of initial levels in two weeks (Figures 1, 2). Penetrated residue declined less rapidly, particularly in Field 1 in which the wrapper leaves were

mature at the time of application.

Endosulfan sulfate increased rapidly as a component of internal residue (Figures 3, 4). The level of endosulfan sulfate in Field 1 continued to increase at 13 days after application.

TABLE I

ENDOSULFAN RESIDUE ON OUTER LEAVES OF HEAD LETTUCE (ppm)

FIELD 1

1.66 qts. Thiodan 2 EC/10 gallons/acre. Applied by air January 13, 1976

Time Post Application	Dislodgeable Residue			Penetrated Residue			Total Residue		
	E.I	E.II	E. Sulfate	E.I	E.II	E. Sulfate	E.I	E.II	E. Sulfate
Preapplication	N.D.	N.D.	0.05	0.26	0.17	0.22	-	-	-
	N.D.	N.D.	0.06	0.27	0.26	0.82	-	-	-
1 Hour	26.3	15.2	N.D.	20.4	12.6	0.70	-	-	-
	19.0	11.9	N.D.	36.4	22.1	0.74	-	-	-
	36.7	21.2	N.D.	28.5	17.8	0.82	-	-	-
20 Hours	9.2	6.5	0.76	24.9	14.8	0.44	38.6	29.1	0.36
	12.	9.2	T	28.4	16.6	0.45			
2 Days	13.9	13.9	0.06	26.2	21.3	0.67			
	15.2	14.0	T	27.8	20.6	0.96			
3 Days	14.2	13.6	N.D.	23.0	16.2	0.91	26.8	23.1	0.79
	7.2	7.4	N.D.	23.8	17.6	0.71			
4 Days	12.4	13.8	N.D.	21.6	17.1	1.3	27.0	26.6	1.3
	11.9	13.7	0.08	20.1	15.1	1.6			
5 Days	5.0	8.3	.07	17.7	17.6	2.0	16.3	18.7	2.4
	6.4	10.7	.11	16.8	16.0	1.9			

FIELD 1 (Continued)

Time Post Application	Dislodgeable Residue			Penetrated Residue			Total Residue		
	E.I	E.II	E. Sulfate	E.I	E.II	E. Sulfate	E.I	E.II	E. Sulfate
6 Days	5.4	7.9	0.08	15.4	16.5	2.3	16.4	21.6	1.8
	2.4	4.6	0.08	17.3	20.0	2.1			
7 Days	5.0	7.7	0.13	17.7	19.1	2.4	12.1	16.3	3.0
	1.8	4.1	T	12.3	14.7	2.0			
8 Days	2.5	6.3	.16	7.4	8.3	1.7	7.2	10.2	2.2
	7.9	13.7	.17	13.4	12.0	2.4			
13 Days	0.84	3.9	T	8.3	13.0	3.7	6.7	9.9	0.60
	1.02	4.2	.11	8.3	10.8	3.9			

FIELD 2

1.6 Pts. Thiodan 4 EC/10 gallons/acre. Applied by air January 16, 1976.

<u>Time Post Application</u>	<u>Dislodgeable Residue</u>			<u>Penetrated Residue</u>			<u>Total Residue</u>		
	<u>E.I</u>	<u>E.II</u>	<u>E. Sulfate</u>	<u>E.I</u>	<u>E.II</u>	<u>E. Sulfate</u>	<u>E.I</u>	<u>E.II</u>	<u>E. Sulfate</u>
Preapplication	0.05	0.05	0.05	0.05	0.05	0.05	-	-	-
1/2 Hour	1.62	0.90	0.05	9.85	4.77	0.14	26.1	13.7	0.18
	3.62	2.45	0.05	22.8	13.0	0.20			
1 Day	3.35	2.69	0.05	15.3	9.13	0.82	14.9	8.94	0.16
	6.90	5.49	0.05	16.7	9.96	0.49			
2 Days	3.79	3.28	0.05	10.4	6.89	0.31	12.7	16.7	0.48
	5.80	5.03	0.05	16.2	9.70	0.60			
3 Days	1.52	1.66	0.05	10.2	7.66	0.95	11.7	9.10	0.81
	1.73	1.80	0.05	12.7	10.0	1.00			
4 Days	1.91	2.92	0.05	12.5	10.6	1.07	10.9	9.39	1.33
	1.68	2.08	0.05	10.5	8.85	1.16			
5 Days	2.35	3.14	0.05	10.8	9.55	1.54	15.2	13.9	1.48
	1.69	2.21	0.05	9.68	8.46	1.52			
10 Days	0.11	0.30	0.05	2.11	1.76	1.11	4.73	6.07	1.42
	0.17	0.49	0.05	2.11	1.90	1.09			
14 Days	*	*	0.05	0.86	1.13	0.69	1.25	1.88	0.91
	*	*	0.05	0.92	1.05	0.77			

T Trace

* Samples lost

FIELD STAFF

James Knaak	- Staff Toxicologist
Charles Kahn	- Agricultural Inspector
Gary Sprock	- Agricultural Inspector
David Conrad	- Agricultural Chemist

Figure 1. Dislodgeable Residue, Field 1

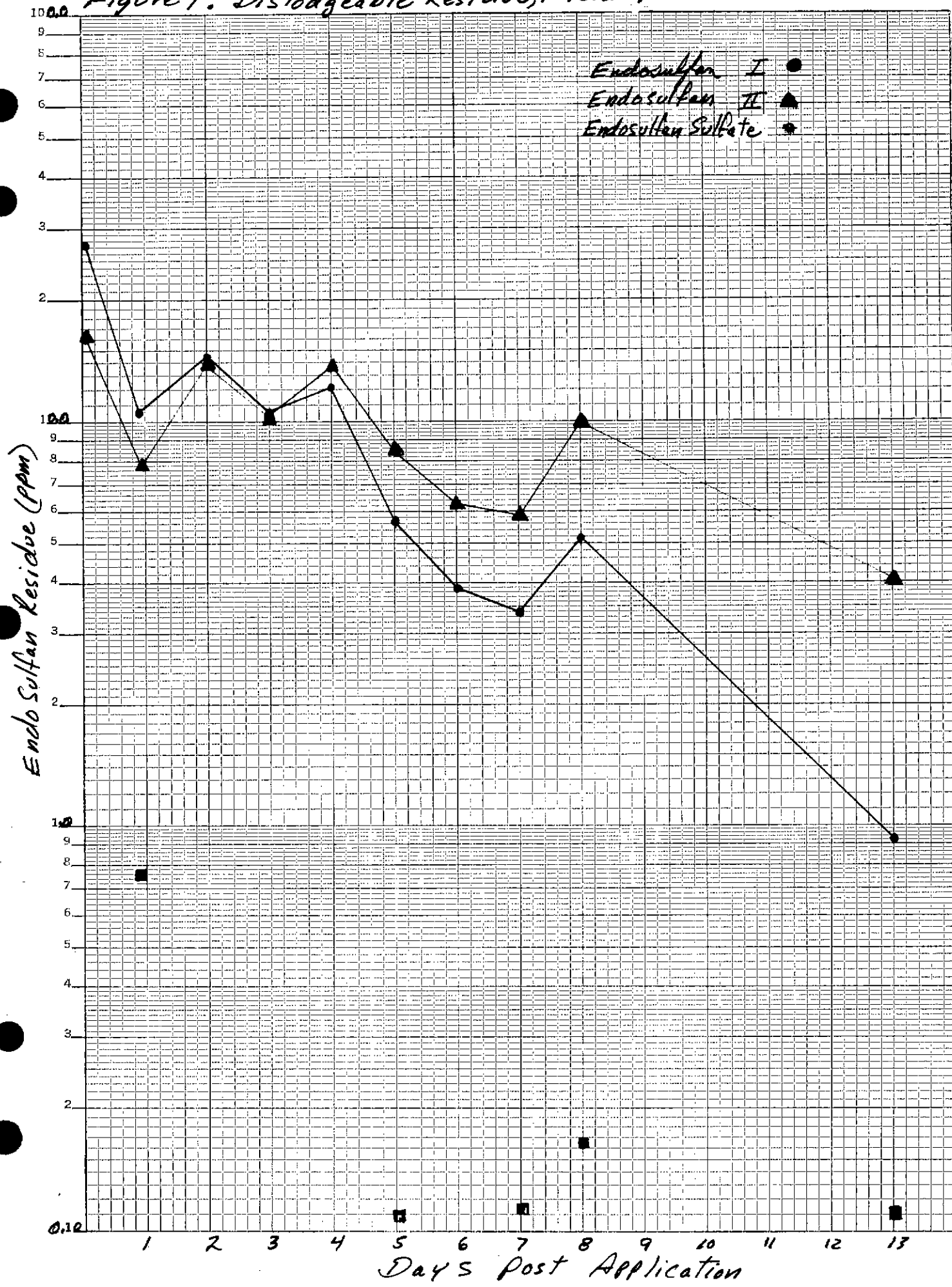


Figure 2. Dislodgeable Residue Field, 2

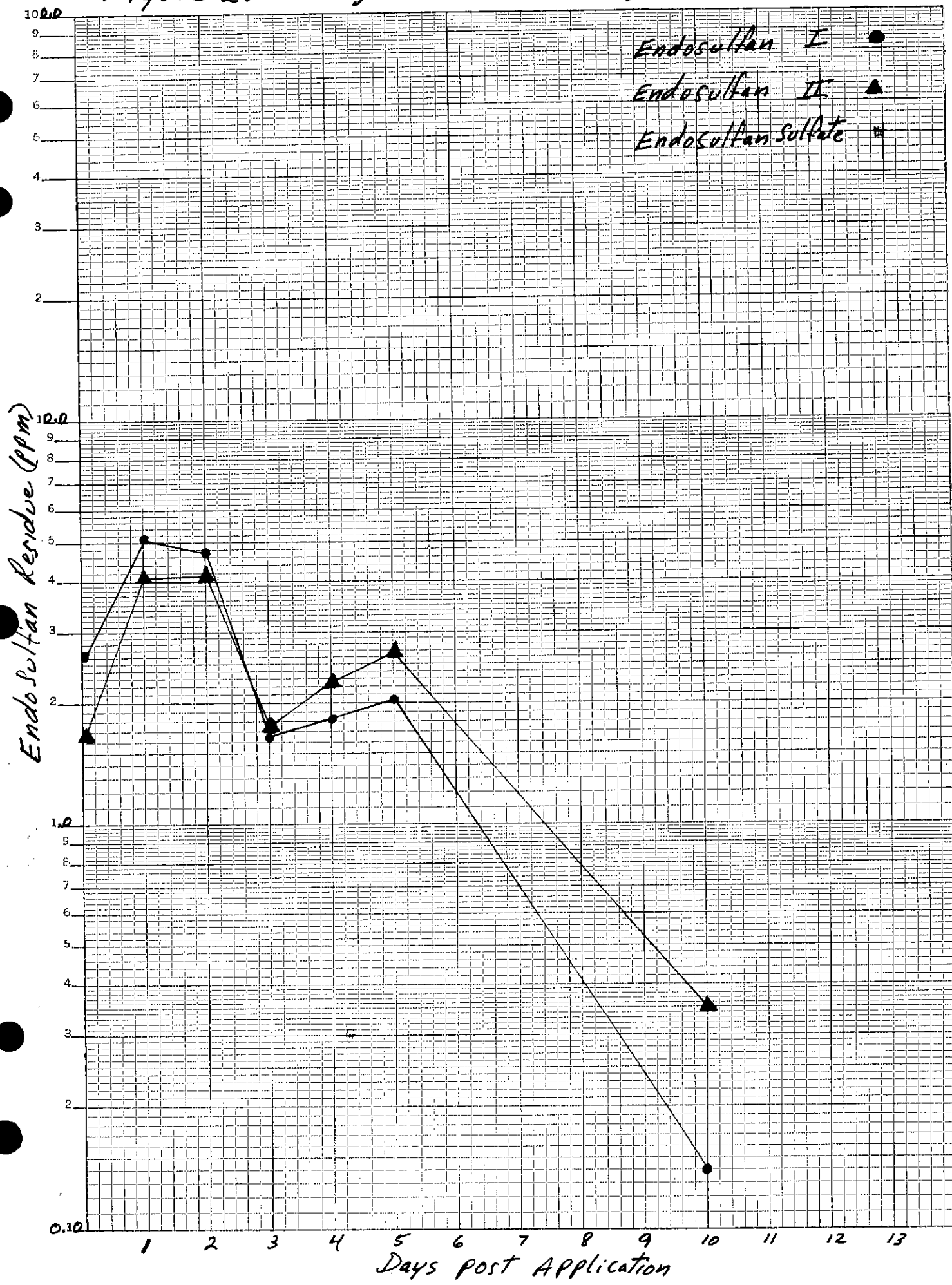


Figure 3. Penetrated Residue, Field 1

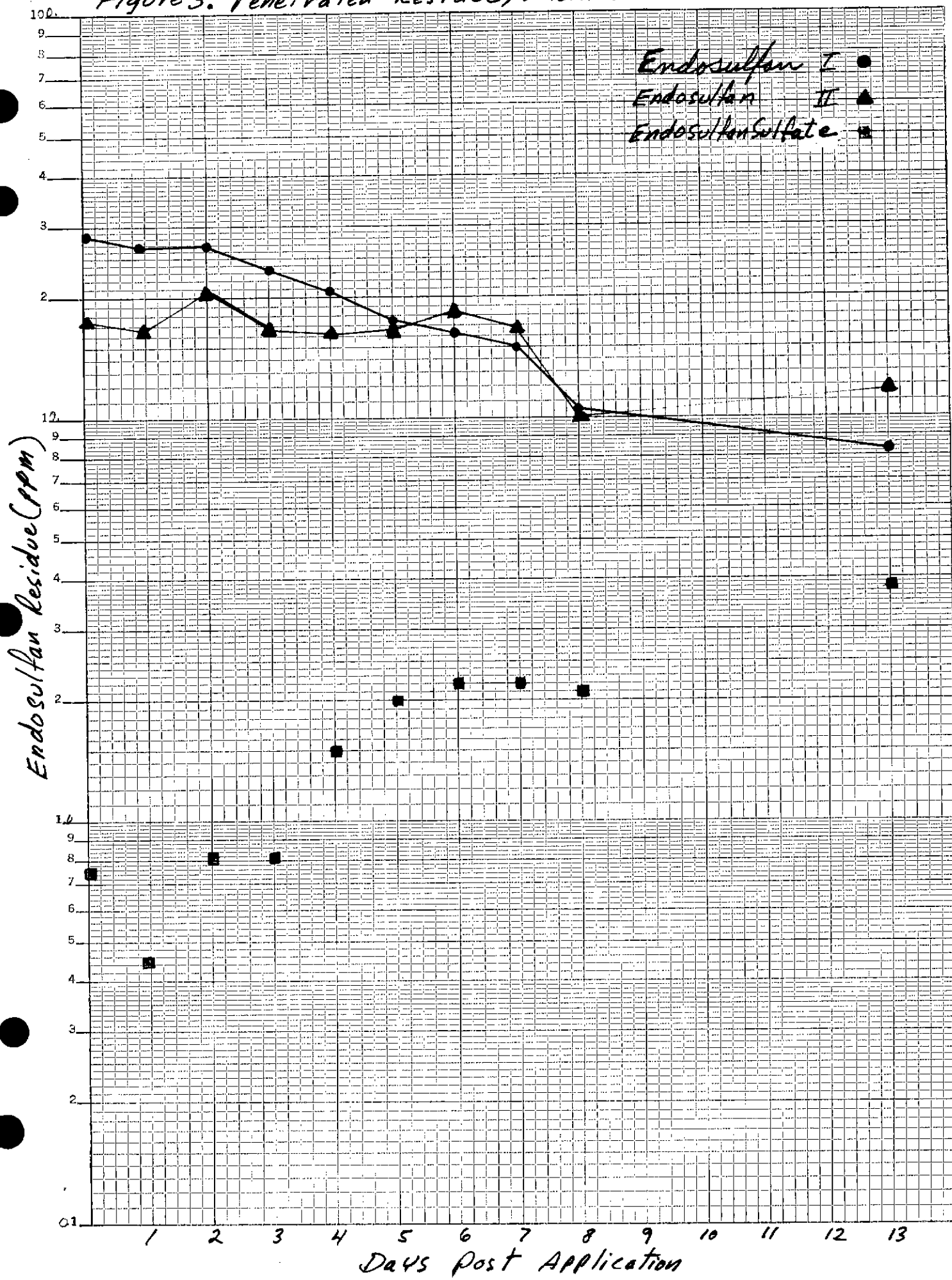
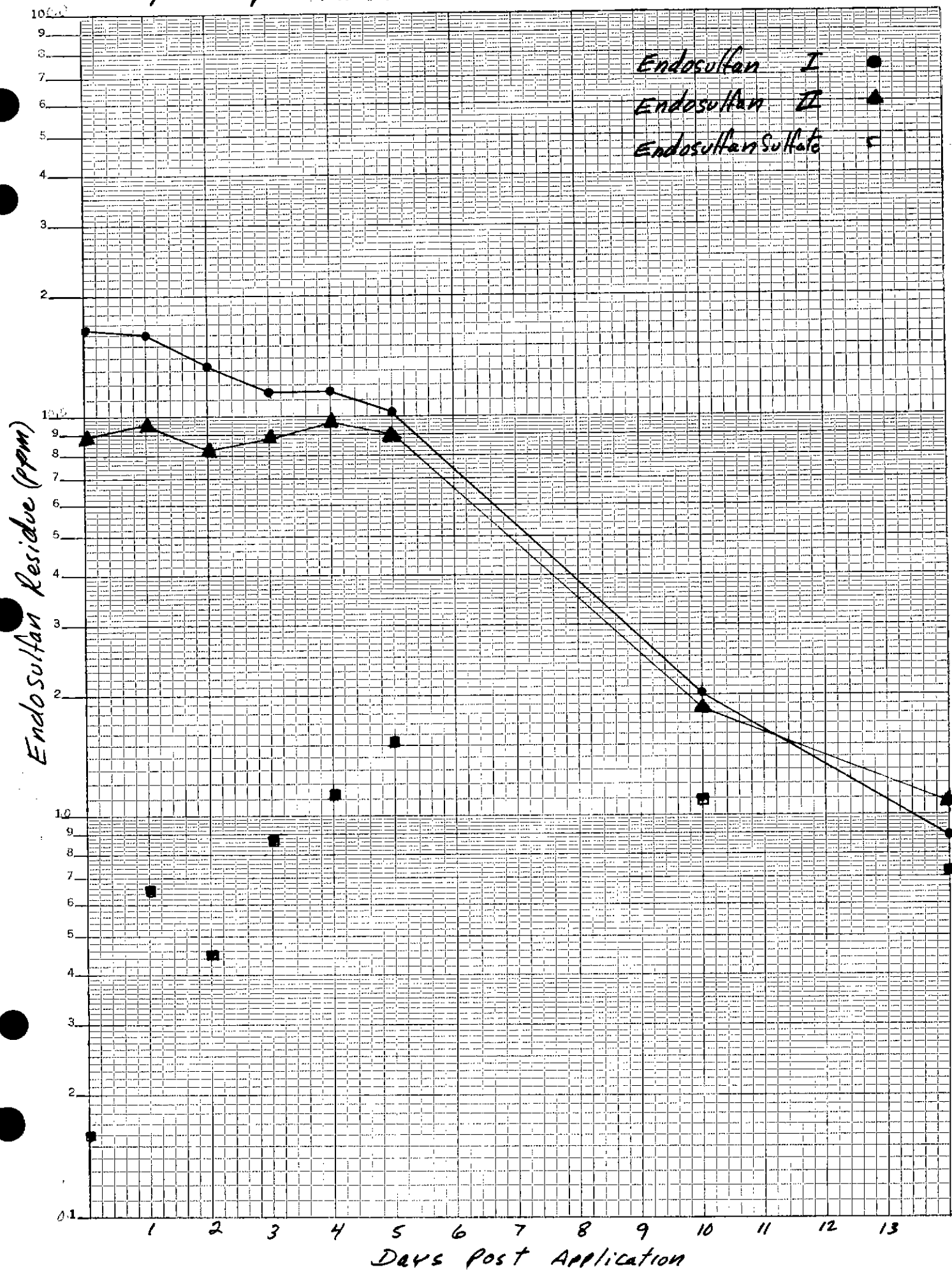


Figure 4. penetrated Residue Field 2



Addendum to HS 220
Recalculation of Dislodgeable Residues

Results of Analysis of Lettuce Foliage
for Dislodgeable Residues of Endosulfan

Sample Interval	Endosulfan Residue (ug/cm ²)		
	E-I	E-II	Sulfate
pre	ND	ND	ND
pre	ND	ND	ND
1-hour	.342	.198	ND
1-hour	.485	.304	ND
1-hour	.505	.292	ND
20-hour	.211	.149	.017
20-hour	.286	.216	ND
2-day	.240	.240	.001
2-day	.272	.250	ND
3-day	.340	.325	ND
3-day	.160	.165	ND
4-day	.174	.193	ND
4-day	.160	.184	.001
5-day	.105	.174	.001
5-day	.119	.198	.002
6-day	.128	.188	.002
6-day	.059	.112	.002
7-day	.130	.200	.003
7-day	.053	.121	ND
8-day	.039	.099	.003
8-day	.124	.214	.003